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INDUSTRIAL HYGIENE SAMPLING AND APPLICATIONS TO AMBIENT SILICA MONITORING

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Interest in ambient exposures to silica has prompted an evaluation of the applicability of the industrial hygiene sampling and analysis experience. Exposure to excessive levels of silica in the workplace has long been recognized as a risk factor for the development of a variety of disabling and sometimes fatal lung diseases. Initial efforts to control occupational exposure to dust were based on reducing exposures as measured by particle-counting techniques. Because silicosis, the disease resulting from exposure to silica, occurs in the lower airways, which can be reached only by small "respirable dust" particles, size selective sampling procedures were introduced for dust monitoring. The analysis of silica in collected dust samples also has undergone development. Initial methods used involved acid digestion of soluble silicates, with subsequent chemical analysis of the insoluble "free silica" fraction. Current methodology relies on the use of X-ray diffraction and infrared technologies to quantify these materials. However, these methods are sensitive to the particle size distribution of the samples. Standard reference materials (SRMs) have been developed for use with respirable size dust samples. Ambient particulate matter is now measured using the U.S. Environmental Protection Agency sampling methods for particulate matter ≤ 10 µm, which approximate the collection efficiency for thoracic fraction samplers. Because the existing calibration SRMs were produced for the measurement of occupational crystalline silica, the need to develop appropriate standards and methods for ambient silica measurements should be evaluated.

INTRODUCTION

The primary National Ambient Air Quality Standards (NAAQS) define levels of air quality that are designed to protect public health within an adequate margin of safety. Secondary NAAQS

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2. Abbreviations: ACGIH, American Conference of Governmental Industrial Hygienists; IR, infrared spectroscopy; mppcf, million particles per cubic foot; MRE, Medical Research Establishment; NAAQS, National Ambient Air Quality Standards; NIOSH, National Institute for Occupational Safety and Health; OSHA, Occupational Safety and Health Administration; PEL, permissible exposure limit; PM_{10}, particulate matter with an aerodynamic diameter less than a nominal 10 µm; SRM, standard reference materials; TLV, threshold limit value; XRD, X-ray diffraction.

3. Key words: dust, exposure limits, respirable, sampling, silica.

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define maximum pollution levels that should protect the public from any known or anticipated adverse effects of a pollutant (40 CFR, 1989). Within this framework, the primary and secondary NAAQS for particulate matter were established based on sampling particles with an aerodynamic diameter less than or equal to a nominal 10 µm (PM_{10}). Recently, ambient exposures to silica have become a focus of concern. Silica long has been recognized as a significant health hazard for workers engaged in mining, sandblasting, construction, and other occupations where significant dust exposures occur. The historical development of dust samplers and the control of dust levels in the occupational environment were motivated by the recognition that exposure to dust containing silica (quartz) produced disabling lung diseases including silicosis (chronic, acute, and accelerated), silico-tuberculosis, and possibly lung cancer (Peters, 1986). Consequently, the evolution of occupational dust limits and the industrial hygiene sampling techniques designed to measure compliance with those standards revolved around monitoring and control of silica dust levels. A variety of devices have been used over the years to quantify workers' exposure to airborne dust levels: filters, impactors, impingers, elutriators, electrostatic and thermal precipitators, and cyclones (Lippmann, 1983; Lioy et al., 1984; Glenn and Kraft, 1986).

The following sections describe the evolution of dust sampling techniques and the workplace exposure limits that have been applied to control occupational exposures, particularly when the dust contained silica. Finally, the application of these principles and techniques to the sampling and analysis of ambient silica levels will be discussed.

INDUSTRIAL HYGIENE DUST SAMPLING AND THE CONTROL OF SILICA

The Greenburg-Smith impinger, developed in 1925 by the U.S. Bureau of Mines, the American Society of Heating and Ventilating Engineers, and the U.S. Public Health Service, was used for more than 30 years as the dust sampling method of choice in mines and factories. A modified, smaller version of the impinger known as the "midget impinger," also was used for measuring the concentration of dust (Mercer, 1973). The midget impinger was designed to be equivalent to the Greenburg-Smith impinger, except that it was much more convenient for field use (Drinker and Hatch, 1954).

Dust particles, collected in the impinger solution, were counted using a counting cell technique adapted from a method used to count plankton in water (Ayer, 1969). The impinger sampler effectively collected dust particles that had an aerodynamic diameter larger than 0.75 µm. The particles were collected by accelerating a dust laden air stream through a nozzle at sonic velocity into a solution of water, ethanol, or mixtures of both (Hall et al., 1965; Jacobson et al., 1970). Concentrations reported from impinger samples usually were stated in million particles per cubic foot of air sampled (mppcf). Rules for counting were not universally standardized. Many industrial hygienists rejected counts of particles with observed diameters larger than 10 µm because these particles are "not respirable" (Lippmann, 1983). Consequently, impinger count data were perceived to be highly inaccurate and subject to training and experience bias among counters (Ayer, 1969). The American Conference of Governmental Industrial Hygienists (ACGIH) recognized that the health risk of exposure to dust was related directly to the total dust concentration and to the concentration of silica in the dust. In 1962, after reviewing available data on dust exposed populations, the ACGIH adopted a threshold limit value (TLV) for dust containing silica (ACGIH, 1986):

$$TLV = \frac{250}{\%Quartz + 5} mppcf. \tag{1}$$

To apply this exposure limit, the concentration of silica (*%Quartz*) was found through chemical analysis of bulk airborne or settled dust samples (Talvitie, 1951, 1964; Talvitie and Hyslop, 1958). The TLV for the work site was then calculated using the above formula. The total dust level was measured using the impinger sampler, and the result obtained was compared to the calculated TLV. For example, if the *%Quartz* was analyzed at 20%, the TLV would be calculated from Equation 1 to be 10 mppcf. Any dust sample for which the measured concentration was over 10 mppcf would exceed the prevailing TLV. In 1970, to make the TLV consistent with a nuisance dust limit of 30 mppcf, the formula was revised to

$$TLV = \frac{300}{\%Quartz + 10} mppcf. \tag{2}$$

For both formulas, the prescribed measurement method was the impinger, coupled with particle count analysis (ACGIH, 1986).

Based on a growing body of literature in the United States (Morrow, 1964) and on the findings of the Johannesburg Pneumoconiosis Conference (Orenstein, 1960), in 1968, the ACGIH proposed a respirable dust TLV based on size selective gravimetric sampling. Using findings from the U.S. Public Health Service, a gravimetric respirable silica TLV of 0.1 mg/m^3 was believed to provide equivalent protection to the existing 10-mppcf total dust concentrations applied to the Vermont granite sheds, where the silica concentration had been measured at 25% (Ayer et al., 1968). Besides the silica TLV, the ACGIH decided to limit size selected respirable dust to 5 mg/m^3. The ACGIH reasoned that the respirable dust TLV would limit the total dust concentration to 15 mg/m^3, based on an assumption that the respirable dust was approximately one-third of the total dust concentration. Using these limits for silica and respirable dust, the ACGIH proposed and adopted a respirable mass TLV formula analogous to the existing particle count formula (Equation 2) for control of respirable dust:

$$TLV = \frac{10}{\%Quartz + 2} mg/m^3. \tag{3}$$

DERIVATION OF THE RESPIRABLE SILICA THRESHOLD LIMIT VALUE FORMULA

The intent of the ACGIH TLV committee was to control silica below 0.1 mg/m^3, while maintaining concentrations of respirable dust below 5.0 mg/m^3. However, the approach they selected to accomplish this also embodied the additive mixture formula. In practice, the additive mixture formula is applied infrequently by industrial hygienists. The additive mixture formula, prescribed for dust mixtures that affect the same organ system with similar pathology is defined by Equation 4:

$$\frac{C_1}{TLV_1} + \frac{C_2}{TLV_2} + \ldots + \frac{C_i}{TLV_i} = 1, \tag{4}$$

where C_i is the concentration of species *i*, and TLV_i is the TLV for species *i* (ACGIH, 1992). When the sum of the terms exceeds unity, the TLV of the mixture is exceeded. Let C_Q be the airborne concentration of silica in milligrams per cubic meter, with a TLV of 0.1 mg/m^3, and let C_D be the airborne concentration of respirable dust in milligrams per cubic meter, with a TLV of 5.0 mg/m^3. The additive-mixture formula, combined with the proposed limits for silica and respirable dust, describes the relationship between C_Q and C_D at their mixture TLV as

$$\left(\frac{C_Q}{0.1}\right) + \left(\frac{C_D}{5.0}\right) = 1. \tag{5}$$

The defining equation for percent silica is

$$\%Quartz = \left(\frac{C_Q}{C_D}\right) \times 100. \tag{6}$$

Multiplying Equation 5 by ($10/C_D$) gives

$$\left\{\left(\frac{C_Q}{C_D}\right) \times 100\right\} + 2 = \left(\frac{10}{C_D}\right). \tag{7}$$

Substituting *%Quartz* from [6] into the term in braces in [7] and rearranging gives

$$C_D = \frac{10}{\%Quartz + 2} mg/m^3. \tag{8}$$

Therefore, the TLV adopted by the ACGIH in 1970 incorporated a silica TLV of 0.1 mg/m^3 and a respirable dust TLV of 5.0 mg/m^3, consistent with the additive mixture formula. The *%Quartz* in the formula was to be found from airborne samples, except where it was decided that other methods were acceptable (Ayer, 1969).

In 1986, the ACGIH adopted a revised TLV for silica that required the direct measurement of respirable quartz with a TLV of 0.1 mg/m^3. The ACGIH also eliminated the "nuisance dust" TLV of 5 mg/m^3 respirable dust, replacing that with a "particles not otherwise classified" limit of 10 mg/m^3 taken as total dust (ACGIH, 1986, 1992). There was no guidance provided by the ACGIH to apply the additive mixture formula to silica and other pneumoconiosis producing dusts. Recent studies by Morrow and co-workers have shown that overloading the lungs with an insoluble dust produced a retardation of macrophage-mediated dust removal leading to impairment of normal lung function (Morrow et al., 1991). These data may provide justification for application of the additive mixture formula when considering combined exposures of coal mine dust, silica, and other respirable dusts.

THE DEFINITION OF RESPIRABLE DUST

The definition of respirable dust has been the focus of many symposia, technical articles, and technical committees. In 1968, the ACGIH established the first size selective TLV for pneumoconiosis producing dusts. The ACGIH proposed a specific definition for respirable dust after considering information on pulmonary deposition in humans. The definition specified several points on the collection efficiency curve, which gives the fraction of particles that pass through a size selective preseparator and are collected on a filter for analysis (Aerosol Technology Committee, 1970; Lippmann, 1970):

Aerodynamic Diameter (microns)	Percent Passing Selector
≤ 2	90
2.5	75
3.5	50
5.0	25
10	0

Another approach to the definition of respirable dust was to define respirable dust as that dust that is collected by a particular instrument. In the United States, that instrument has been the 10-mm nylon cyclone. Because the 10-mm nylon cyclone's collection efficiency curve shifts with changes in the operating flow rate, there has been controversy about whether the flow rate should be 1.4, 1.7, or 2.0 L/min (Bartley and Breuer, 1982; Caplan et al., 1977; Ettinger et al., 1970; Knuth, 1969; Tomb and Raymond, 1970; Tomb and Treaftis, 1976). After examining the flow rate bias curves, which measure the difference between the ACGIH respirable dust deposition curve and the sampler's observed performance, the ACGIH decided that 1.7 L/min should be used for sampling respirable dust. The National Institute for Occupational Safety and Health (NIOSH) has not adopted a particular definition of respirable dust. However, the NIOSH Manual of Analytical Methods, in Method 0600 ("Nuisance Dust, Respirable"), recommends using the 10-mm nylon cyclone at a sampling flow rate of 1.7 L/min (NIOSH, 1984).

FEDERAL REGULATION OF OCCUPATIONAL EXPOSURE TO SILICA

In coal mines, when silica is present at a concentration greater than 5%, the 2.0 mg/m^3 respirable dust standard (*RDS*) is reduced according to the formula (30 CFR, 1989)

$$RDS = \frac{10}{\%Quartz}\, mg / m^3 . \qquad (9)$$

The enforcement procedure is to collect as few as one respirable silica sample on a working section to find the *%Quartz* that will apply for that location. If the *%Quartz* is greater than 5%, then the section must maintain an *RDS* below that computed using Equation 9. Note that the *RDS* samples are collected using a 10-mm nylon cyclone operating at 2.0 L/min, and the collected mass is multiplied by a 1.38 correction factor. This factor is intended to convert the cyclone measured dust concentration to an equivalent measurement that would have been made with a Medical Research Establishment (MRE) horizontal elutriator (Tomb and Raymond, 1970). The conversion is necessary because the U.S. respirable coal mine dust standard was based originally on British epidemiological data, where the dust concentrations were measured using the MRE instrument. The collection efficiency of a 10-mm nylon cyclone shifts when operated at 2.0 L/min, collecting fewer large particles compared to operation at 1.7 L/min. Because the size selection differs, and because the 1.38 correction factor is applied to the measured concentrations, silica sampling data from coal mine dust compliance sampling programs are not directly comparable to results obtained in other industries and for noncoal mines where the 1.7-L/min sampling flow rate is used routinely.

Prior to a 1992 Appeals Court ruling, the Occupational Safety and Health Administration (OSHA) permissible exposure limit (PEL) for silica in nonmining industrial workplaces was 0.1 mg/m^3, measured as respirable dust, using a 10-mm nylon cyclone operated at 1.7 L/min (29 CFR, 1989; OSHA, 1991). Because of that legal ruling, the present OSHA silica dust standard is 1970 ACGIH formula—Equation 8. The NIOSH recommended exposure limit for silica is 0.05 mg/m^3, measured as respirable dust, and NIOSH notes that silica inhalation presents a potential lung cancer risk (NIOSH, 1974, 1984, 1992).

Recently, the ACGIH proposed a slight change in the definition of respirable dust. The definition adopted in 1968 was based on a 50% collection efficiency for particles with an aerodynamic diameter of 3.5 µm. The current proposed definition recommends a 50% collection efficiency for particles with an aerodynamic diameter of 4.0 µm (Soderholm, 1991; ACGIH, 1992). The motivation behind this slight revision to the definition of respirable dust was to bring the ACGIH definition into agreement with the international definition of respirable dust being adopted by the International Standards Organization (Soderholm, 1989). Their proposed definition is intended to apply to health related air sampling in both workplaces and the general environment (ISO, 1991).

OTHER SIZE SELECTIVE SAMPLING CRITERIA FOR OCCUPATIONAL ENVIRONMENTS

Besides the definition of respirable dust, two other mass fractions for dust sampling have been defined: (1) the inhalable (or inspirable) particulate mass and (2) the thoracic particulate mass (ISO, 1981). The inspirable fraction describes those particles that can be inhaled and includes all particles that may deposit in the head, the tracheobronchial tree, and the alveolar (gas exchange) region of the lungs (Soderholm, 1984). The thoracic fraction is defined by a cumulative lognormal function with a median (i.e., 50% collection efficiency) of 10.0 µm and a geometric standard deviation of 1.5 (Rabbe, 1984). This definition includes those particles small enough to penetrate beyond the nasal/pharyngeal passages of the head and also includes particles that deposit in the tracheobronchial tree and in the alveolar region of the lung.

Each of the three definitions describes "penetration" curves for particles based on their aerodynamic diameters. Particles with aerodynamic diameters much smaller than the median cut point are collected with almost 100% efficiency. Samplers designed to measure penetration do not measure "deposition" in any particular region of the lung. Alveolar deposition is less than 100% for particles between 0.1 and 1.0 µm aerodynamic diameter because many particles in this size range are subsequently exhaled (Chan and Lippman, 1980; Stahlhoffen et al., 1980). Therefore, respirable dust samplers will overestimate the fraction deposited in the alveolar region of the lung for submicrometer size particles. Tracheobronchial deposition is not measured by the thoracic fraction samplers because those samplers include and collect particles that would be deposited in the alveolar region (i.e., the respirable fraction). Extrathoracic deposition (the fraction deposited in the head and pharynx, down to and including the larynx [i.e., the "nasal/pharyngeal" fraction]) is not measured with an inspirable or inhalable fraction sampler because that fraction includes particles that would penetrate further and be deposited in the tracheobronchial and alveolar regions of the lung (Raabe, 1984).

AMBIENT SAMPLING USING THE PM_{10} SAMPLING SYSTEM

The U.S. Environmental Protection Agency specified in its primary and secondary NAAQS for particulate matter, that particulate matter shall be measured in the ambient air as PM_{10} particles, with an aerodynamic diameter less than or equal to a nominal 10 µm (40 CFR, 1989). The requirements for a PM_{10} sampler were specified similar to the thoracic fraction sampling criteria proposed by Chan and Lippmann (Chan and Lippmann, 1980; Kashdan et al., 1986; Liu and Pui, 1986).

The selection of an appropriate sampling system should be based on the criteria that the measured quantities are related to the risk being controlled (Roach, 1953; Ayer, 1969). The selection of one or more of the size selective criteria could be guided by the following classification scheme.

(1) Respirable dust sampling—suggested when the aerosol is insoluble and the disease is associated with the gas exchange regions of the lungs, as in fibrotic diseases like silicosis or coal workers' pneumoconiosis, or emphysema.

(2) Thoracic dust sampling—suggested when the aerosol is believed to produce asthma, to aggravate chronic bronchitis, or to be responsible for inducing bronchogenic tumors. Thoracic sampling is appropriate if the material that deposits in the head airways does not contribute to its toxicity.

(3) Inhalable or inspirable dust—suggested when the aerosol is likely to be absorbed by the body wherever it deposits, or if the dust is associated with nasal cancer as in woodworkers (ISO, 1981; Stuart et al., 1984).

For environmental particulate exposures, concern should be based on health effects including asthma, bronchitis, emphysema, fibrosis, and pneumoconiosis. Based on these broad needs, the selection of PM_{10} sampling for the NAAQS for particulate matter is appropriate. The most appropriate size selective criterion for measuring silica concentrations, exclusive of other particulate exposures, would be the respirable criterion that has a nominal median aerodynamic diameter of 3.5–4.0 µm. If silica were uniformly distributed, without regard to particle size, then thoracic sampling (PM_{10}) would provide an acceptable surrogate for respirable mass sampling. However, the concentration of silica is often higher in the larger size fractions (Davis et al., 1984; EPA, 1996). It has been suggested that this may be because quartz, which is harder than most minerals, resists comminution to fine particle sizes (Ayer, 1969).

PARTICLE SIZE DEPENDENCE IN ANALYSIS

There are three principal analytical methods that have been used for analysis of silica particulate. The oldest of the methods is based on the differential solubility of crystalline silica with various silicates and amorphous silica, when treated with hot phosphoric acid. Following the dissolution of the nonsilica containing materials, the remaining "free silica" was quantified either by weighing the residue (Talvitie, 1951) or, more precisely, by colorimetric determination (Talvitie and Hyslop, 1958; Talvitie, 1964). In an attempt to find suitable alternatives to the Talvitie method, investigators examined other spectral methods such as infrared spectroscopy (IR) and X-ray diffraction (XRD). One study examined particle counting using a light microscope with dispersion staining (Edwards, 1965). In a comparative study of IR and XRD methods, it was found that both IR and XRD could quantify silica particles with approximately equal precision and ruggedness (NIOSH, 1983). However, both methods were subject to interference by other common minerals, and both exhibited a response dependent on the particle size of the sample. When a pure silica standard (Min-U-Sil®) was used to establish a calibration curve for the XRD method, the instrumental response for the 10-µm Min-U-Sil® was 1.6 times the response obtained for the 5 µm Min-U-Sil® (NIOSH, 1983). Similarly, it was observed for the IR method that the observed absorbance peaks varied by as much as 30% for particle diameters varying from 0.65–5.8 µm (Lorberau, 1989). Because of these response characteristics of the methods, the choice of a suitable reference standard was critical to making accurate measurements of silica concentrations (Verma et al., 1992). To improve the standards for XRD analysis of silica, the National Bureau of Standards produced a standard reference material (NBS-SRM#1878) by cycloning Min-U-Sil® 5, an α-quartz sold by the U.S. Silica Company, Berkeley Springs, West Virginia. The particle size distribution of the standard reference material approximated that found on respirable dust samples, thus reducing systematic bias from particle size effects on instrument response.

DISCUSSION

Sampling techniques used for monitoring occupational exposure to silica may be applicable to monitoring ambient particulate concentrations. The key factors that must be considered include the levels of exposure to be quantified, and the capability to modify existing sampling and analytical techniques for airborne particles in the ambient environment. Industrial hygiene sampling systems typically are designed to collect full shift, 8-h , time-weighted-average samples, for comparison to an OSHA PEL for silica exposures below 0.1 mg/m^3 (100 $\mu g/m^3$). Conversely, the NAAQS for particulate matter are stated in terms of 24-h average concentrations, with a primary standard of 150 $\mu g/m^3$ and a secondary standard of 50 $\mu g/m^3$. The control concentration, or exposure limit, is typically lower for the ambient exposure than for the OSHA PEL promulgated for industrial hygiene purposes. Appropriate sampling systems for measuring the ambient silica concentration will need to consider the sensitivity of the available methods to quantify the low dust concentrations likely to be found in the ambient samples. These methods will also need to correct for interferences from other minerals and dusts that also will be collected by the ambient samplers. Finally, the potential bias introduced through the particle size dependence of the analytical methods may be minimized by developing standard reference materials that match the size selective criteria used in ambient particulate matter sampling.

The appropriate size fraction for health related sampling of silica is the respirable fraction. Field studies, including particle size distribution measurements and side-by-side sampling with PM_{10} and respirable dust samplers, are needed to evaluate these samplers' performance. These studies should determine if the PM_{10} system correlates sufficiently with the respirable fraction for PM_{10} to be used as a surrogate, or whether respirable dust samples must be collected separately.

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